

Sertifikaat

PATENTKANTOOR

REPUBLIC OF SOUTH AFRICA

DEPARTEMENT VAN HANDEL
EN NYWERHEID



10/532891 #2
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Certificat
PATENT OFFICE
REPUBLIEK VAN SUID-AFRIKA

DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

1B/03/4788

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| REC'D 27 NOV 2003 | |
| WIPO | PCT |

the documents attached hereto are true copies of
the Forms P2, P6, provisional specification and
drawings of South African Patent Application No.
2002/8731 in the name of Element Six (Proprietary)
Limited

Filed : 29 October 2002

Entitled : Composite Material

**PRIORITY
DOCUMENT**

SUBMITTED OR TRANSMITTED IN
COMPLIANCE WITH RULE 17.1(a) OR (b)

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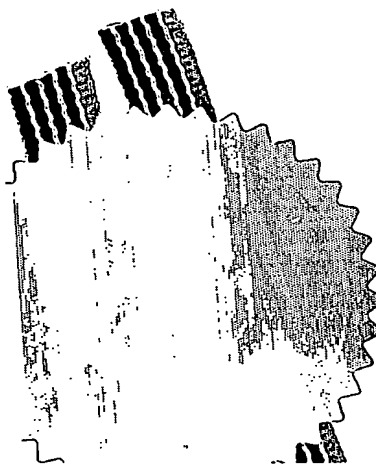
PRETORIA

in die Republiek van Suid-Afrika, hierdie
in the Republic of South Africa, this

13th

dag van
day of

November 2003




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Registrateur van Patente

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
APPLICATION FOR A PATENT
AND ACKNOWLEDGEMENT OF RECEIPT 28.10.02
(Section 30 (1) - Regulation 22)

REPUBLIC OF SOUTH AFRICA FORM P.1

REVENUE

R 0060.00

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

HASR 711

INFORMS

REPUBLIC VAN SUID AFRIKA
S & F REFERENCE

OFFICIAL APPLICATION NO.

| | | |
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| 21 | 01 | 2002/8737 |
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PA133718/P

FULL NAME(S) OF APPLICANT(S)

| | |
|----|-----------------------------------|
| 71 | ELEMENT SIX (PROPRIETARY) LIMITED |
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ADDRESS(ES) OF APPLICANT(S)

| |
|--------------------------------------------------------------------------------------------------------------|
| SEO BUILDING, CORNER CROWNWOOD & BOOYSENS RESERVE ROADS, THETA, JOHANNESBURG, 2001, GAUTENG, SOUTH AFRICA |
|--------------------------------------------------------------------------------------------------------------|

TITLE OF INVENTION

| | |
|----|--------------------|
| 54 | COMPOSITE MATERIAL |
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THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

| | | |
|--------------|-------------|-----------|
| COUNTRY: NIL | NUMBER: NIL | DATE: NIL |
|--------------|-------------|-----------|

THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

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| 21 | 01 | |
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THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

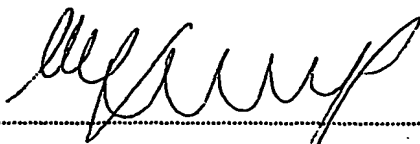
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THIS APPLICATION IS ACCOMPANIED BY:

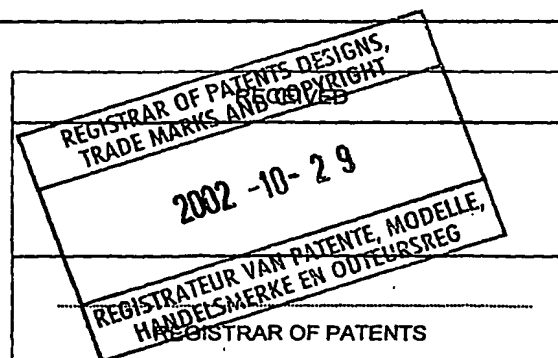
- ☒ 1. A single copy of a provisional specification of 13 pages.
- ☒ 2. Drawings of 2 sheets.
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No.
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

74 ADDRESS FOR SERVICE: SPOOR & FISHER, SANDTON

Dated: 29 October 2002



SPOOR & FISHER
PATENT ATTORNEYS FOR THE APPLICANT(S)



| REPUBLIC OF SOUTH AFRICA | | REGISTER OF PATENTS | | PATENTS ACT, 1978 | | |
|-----------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------|---------------------------|--------------------|-------------------|-----|--|
| OFFICIAL APPLICATION | | LODGING DATE: PROVISIONAL | | ACCEPTANCE DATE | | |
| 21 | 01 | 22 | 29 OCT 2002 | 47 | | |
| INTERNATIONAL CLASSIFICATION | | LODGING DATE: COMPLETE | | GRANTED DATE | | |
| 51 | | 23 | | | | |
| FULL NAME(S) OF APPLICANT(S)/PATENTEE(S) | | | | | | |
| 71 | ELEMENT SIX (PROPRIETARY) LIMITED | | | | | |
| APPLICANTS SUBSTITUTED: | | | | DATE REGISTERED | | |
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| ASSIGNEE(S) | | | | DATE REGISTERED | | |
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| FULL NAME(S) OF INVENTOR(S) | | | | | | |
| 72 | 1. SIGALAS, IAKOVOS 2. CHAPMAN, RAYMOND ALBERT 3. DAVIES, GEOFFREY JOHN 4. MASETE, MOSIMANEGAPE STEPHEN | | | | | |
| PRIORITY CLAIMED | | COUNTRY | | NUMBER | | |
| N.B. Use International abbreviation for country (see Schedule 4) | | 33 | NIL | 31 | NIL | |
| DATE | | 32 | NIL | | | |
| TITLE OF INVENTION | | | | | | |
| 54 | COMPOSITE MATERIAL | | | | | |
| ADDRESS OF APPLICANT(S)/PATENTEE(S) | | | | | | |
| SEO BUILDING, CORNER CROWNWOOD & BOOYSENS RESERVE ROADS, THETA, JOHANNESBURG, 2001, GAUTENG, SOUTH AFRICA | | | | | | |
| ADDRESS FOR SERVICE | | | | S & F REF | | |
| 74 | SPOOR & FISHER, SANDTON | | | PA133718/P | | |
| PATENT OF ADDITION NO. | | | DATE OF ANY CHANGE | | | |
| 61 | | | | | | |
| FRESH APPLICATION BASED ON | | | DATE OF ANY CHANGE | | | |
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REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978

PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

LODGING DATE

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| 21 | 01 | 2002/8731 |
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| 22 | 29 OCTOBER 2002 |
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FULL NAMES OF APPLICANTS

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| 71 | ELEMENT SIX (PROPRIETARY) LIMITED |
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FULL NAMES OF INVENTORS

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| 72 | SIGALAS, IAKOVOS CHAPMAN, RAYMOND ALBERT DAVIES, GEOFFREY JOHN MASETE, MOSIMANEGAPE STEPHEN |
|----|------------------------------------------------------------------------------------------------------|

TITLE OF INVENTION

| | |
|----|--------------------|
| 54 | COMPOSITE MATERIAL |
|----|--------------------|

BACKGROUND OF THE INVENTION

This invention relates to a composite material, a method of making the composite material and a method of making a tool component.

Tool components utilising diamond compacts, also known as PCD; and cubic boron nitride compacts, also known as PCBN, are extensively used in drilling, milling, cutting and other such abrasive applications. The tool component will generally comprise a layer of PCD or PCBN bonded to a support, generally a cemented carbide support. The PCD or PCBN layer may present a sharp cutting edge or point or a cutting or abrasive surface.

PCD cutters are well-known and widely used in drill bit technology as the cutting element in drill bits used in core drilling, oil and gas drilling, and other similar applications. Such cutters generally comprise a PCD table formed on a hard metal substrate by a high temperature and high pressure sintering process. The substrate is then either brazed on an elongated support, or is directly brazed in a pocket of the drill bit, in a manner that exposes the PCD table to the surface for cutting.

It is known that PCD cutters inherently have residual stresses due to the mismatch of the properties of PCD to those of the substrate. The relevant properties in this context are the thermal expansion coefficient and the elastic moduli and compressibilities of the two materials. These stresses are

particularly pronounced at the interface, but are present mostly throughout the cutter. These stresses tend to be compressive within the PCD layer and tensile within the substrate. However tensile stresses do exist within the PCD layer, particularly in cases where a non-planar interface is used. These stresses can combine with the applied stresses during the rock drilling process and bring about the fracture of the cutter.

Furthermore, such stresses are known to increase in magnitude during the brazing process used to attach the cutter to the drill bit. This increase in stress can cause fracture of the PCD layer or of the substrate, even without the application of an external stress.

Various solutions have been suggested in the art for modifying the residual stresses in PCD cutters in order to avoid such failures. For example, it has been suggested that configuring the diamond table and/or carbide substrate in a particular way may redistribute the stress such that tensile stresses are reduced, as disclosed in U.S. Pat. No. 5,351,772 to Smith, and U.S. Pat. No. 4,255,165 to Dennis. Other cutter configurations, which reduce residual stresses, are disclosed in U.S. Pat. No. 5,049,164 to Horton; U.S. Pat. No. 5,176,720 to Martell et al.; U.S. Pat. No. 5,304,342 to Hall; and U.S. Pat. No. 4,398,952 to Drake. Methods for relieving residual stresses by back-grinding the substrate, annealing, or by varying the properties of the substrate are disclosed in U.S. Pat. No. 6,220,375.

U.S. Pat. No. 4,604,106 to Hall *et al* discloses the use of precemented carbide particles in a PCD matrix in order to introduce a graded interface between the PCD table and the carbide substrate. A similar material is also disclosed in U.S. Pat. No. 4,525,178. Although this approach gives efficient grading of properties, it requires the preparation of carbide particles by crushing, which can be an expensive process. Furthermore such materials are known to chip, because the random disposition of the precemented carbide particles in the

PCD matrix carries with it the possibility of various agglomerates forming in the body of the material, thus increasing its flaw size, and consequently reducing its strength.

U.S. Pat. No. 5,370,195 discloses drill bit inserts comprising a number of layers positioned between the substrate and the outer PCD layer. These intermediate layers are essentially diamond-carbide composites. Each composite is made out of individual diamond crystals mixed with tungsten carbide, or titanium carbide, or titanium carbonitride particles. Such materials are useful in managing the residual stresses in the drill bit inserts, but possess inferior strength and toughness, due to the poor adhesion of the diamond particles to the binder phases used.

Use of interfaces as a means of managing the residual stresses in a PCD cutter requires that the interface material has good wear resistance, that would be equal or slightly inferior to that of the PCD layer, and equal or better than that of the carbide substrate. This arrangement would ensure that during the cutting action a lip is formed below the PCD table, allowing for the concentration of stress at the cutting point, thus ensuring fracture of the rock being cut. If the wear resistance of the interlayer was less than that of the carbide support, the wear of the interlayer would be excessive, the PCD lip would lose support during the cutting action, and it would break. If the wear resistance of the interlayer is too high, the lip that develops in use is too shallow and the cutting action is not substantially improved. If the wear resistance of the interlayer is too low, the lip that develops is too deep and the PCD layer is not afforded sufficient support and the cutting edge fails prematurely. Therefore, there is an optimum relationship between the wear resistance of the PCD, the interlayer and that of the substrate.

U.K. Pat. No. 2, 326, 655 discloses the use of PCD granules in a carbide skin. These granules are then used to make a material that has good wear

resistance and toughness, which is suitable as a wear part or as an interface material. Such a material relies on the tensile stresses generated in the carbide phase to cause cracking to run through this phase, thus improving this material's fracture toughness. In order to ensure that the crack does indeed run through the carbide phase, the grade chosen is fine grained and relatively brittle. Thus, no major improvement in toughness can be attained.

SUMMARY OF THE INVENTION

According to one aspect of the invention, a composite material comprises a plurality of cores of material selected from the group comprising carbides, nitrides, carbonitrides, cemented carbides, cemented nitrides, cemented carbonitrides and mixtures thereof, dispersed in a matrix, the matrix comprising the components for making an ultra-hard material and a suitable binder.

The ultra-hard material is polycrystalline in nature and is typically PCD or PCBN.

The cores are typically provided as individual particles or in the form of granules.

According to a further aspect of the invention, a method of producing a composite material as described above includes the steps of:

- (i) providing a plurality of cores of material selected from a group comprising carbides, nitrides, carbonitrides, cemented carbides, cemented nitrides, cemented carbonitrides and mixtures thereof;

- 5 -

- (ii) providing the components for making an ultra-hard material and a suitable binder; and
- (iii) consolidating the cores, components and binder to produce a composite material.

According to yet another aspect of the invention, a method of producing a tool component includes the steps of:

- (i) providing a substrate;
- (ii) providing a composite material as described above;
- (iii) placing a layer of the composite material on a surface of the substrate to produce an unbonded component; and
- (iv) subjecting the unbonded component to conditions of elevated temperature and pressure suitable to produce an ultra-hard material.

According to yet another aspect of the invention, a method of producing a tool component includes the steps of:

- (i) providing a substrate;
- (ii) providing a composite material as described above;
- (iii) placing a layer of the composite material on a surface of the substrate;

- (iv) placing a layer of the components for making an ultra-hard material on the layer of composite material to produce an unbonded component; and
- (v) subjecting the unbonded component to conditions of elevated temperature and pressure to produce an ultra-hard material from the components.

The cores are typically provided as granules coated with the components for making an ultra-hard material and the binder.

The granules may be further coated with a second coating comprising material selected from the group comprising carbides, nitrides, carbonitrides, cemented carbides, cemented nitrides, cemented carbonitrides, and mixtures thereof, or the components for making an ultra-hard material of a different grade to that of the first coating.

The composite may be a moulded composite which takes on the shape of the surface of the substrate on which it is placed and/or the shape of a surface of the ultra-hard material layer. In this regard, the composite may be pre-cast in the appropriate form or, alternatively, moulded *in situ*.

The composite material typically takes on a honeycomb structure of a hard material and cores within the pores of the honeycomb structure bonded to the honeycomb structure. The pores of the honeycomb structure may be ordered or random.

The components necessary to produce an ultra-hard material may comprise a mass of ultra-hard abrasive particles and optionally a second phase comprising a solvent/catalyst or a precursor to a solvent/catalyst, in particulate form, for the ultra-hard abrasive particle. Such components may include the

superalloys, such as the Nimonic® and Stellite® alloys, and high temperature brazes.

The sintering conditions in step (iv) or step (v), as the case may be, are such that the ultra-hard abrasive particles are crystallographically stable.

The particles in the core and, where appropriate, in the coating, are provided in a suitable binder such as an organic binder. This binder will preferably be removed prior to the sintering of step (iv) or (v). Examples of suitable binders include but are not limited to camphor, methyl cellulose and polyethylene glycol.

The plurality of granules may be consolidated by applying pressure to the granules, for example, in a confined space such as a die. The consolidated composite is a green state product which has coherency, but which may also be severed, for example, by cutting. A piece which may be severed and removed from the consolidated or coherent composite has flexibility and may be applied to surfaces which are flat or profiled, e.g. a curved surface. The die for consolidating the granules may be provided with one or both punches profiled such that the green state product has at least one surface that has a shape complementary to the substrate upon which it is to be placed. In this case, the composite layer presents a working surface or a cutting edge. The other surface thereof may also be profiled to accommodate a further layer such as an ultra-hard material layer or another layer of a similar composite material but of a different composition, for instance in terms of the ultra-hard material content or in the grade of the ultra-hard material, presenting a working surface or cutting edge. In this case, the composite material provides an interlayer between the substrate and the cutting layer. In order to provide for a grading of properties, several interlayers of composite material having different compositions may be provided.

Where the cores of the composite material are formed from carbide particles, these will typically be tungsten carbide particles, titanium carbide particles, tantalum carbide particles or molybdenum carbide particles. The metal binder may be any metal binder known in the art such as iron, nickel, cobalt or an alloy containing one or more of these metals.

The substrate will typically be a cemented carbide substrate.

The granules may be produced by providing a core and then coating the core with an ultra-hard material in the presence of an organic binder. Coating may take place by fluidising the cores and spraying the coating thereon or by pelletising in a pan.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will now be described in more detail, by way of example only, with reference to the accompanying drawings in which:

- Figure 1** is a cross section through a granule used in making a composite material of the invention;
- Figures 2 & 3** illustrate schematically the consolidation of granules to form a composite material of the invention;
- Figure 4** is a cross-section through an alternative granule for making a composite material of the invention; and
- Figure 5** is an exploded cross-section through a tool component of the invention.

DESCRIPTION OF EMBODIMENTS

Referring to Figure 1, a granule 10 comprises a core 12 and a coating 14 substantially enclosing the core 12. The granule 10 illustrated is of uniform shape and spherical. The granule does not have to be of such uniform shape, nor need it be spherical. Other shapes are possible. For convenience, this embodiment of the invention will be described with reference to carbide particles. It is to be understood, however, that the cores can comprise other core material selected from the group comprising nitrides, carbonitrides, cemented carbides, cemented nitrides, cemented carbonitrides and mixtures thereof.

The core 12 comprises a mixture of carbide particles and binder metal in particulate form.

The coating 14 comprises ultra-hard abrasive particles such as diamond or cubic boron nitride and optionally a metal or precursor in particulate form. Such metal may be a solvent/catalyst or another metal which will sinter under the applied conditions of temperature and pressure.

An organic binder such as methyl cellulose is present in both the core 12 and the coating 14 and provides both the core 12 and the coating 14 and the granule 10 as a whole with coherency. Other non-limiting examples of the organic binder include camphor and polyethylene glycol.

A plurality of the granules 16, as illustrated by Figure 2, are placed in a container 18. Pressure in the direction of the arrows 20 is applied to the granules 16 causing them to consolidate into a composite material as illustrated by Figure 3. The composite material comprises a plurality of cores 22 in what is now a matrix 24 produced from the coatings 14. Although so-

called uniaxial compression is described in this embodiment pressure may be applied from above and below the granules 16 or isostatically.

In an alternative embodiment of the invention, as shown in Figure 4, the granule 10 includes a second coating 26 substantially covering the coating 14. The coating 26 can be formed from the same material as the core 12, or it can be made of the same material as the coating 14, but of a different grade in order to allow for a grading of properties.

A layer or portion 28 of the composite material is severed along line 30, as shown in Figure 3, and removed from the composite material.

The portion or layer 28 has flexibility and may be placed on the surface 32, in this case an irregular surface, of a substrate 34, preferably a cemented carbide substrate, or between the surface 32 of the substrate 34 and a surface 36, in this case an irregular surface, of an abrasive layer 38, as shown in Figure 5.

The green state product of Figure 5 is placed in a suitable capsule for insertion into the reaction zone of a conventional high temperature/high pressure apparatus. The organic binder is first removed by heating the capsule to drive off the binder. The capsule is then placed in the reaction zone and the contents of the capsule subjected to conditions of elevated temperature and pressure such that the ultra-hard abrasive is not degraded. Such conditions may be those at which the ultra-hard abrasive is crystallographically stable. This has the effect of producing cemented carbide out of the material of core 10 and an abrasive compact out of the material of coating 14. The abrasive compact will be bonded to the cemented carbide. The layer 28 will be bonded to the surface 32 of the substrate 34 and the surface 36 of the abrasive compact 38 producing a tool component.

Although the use of coated granules to form the composite material is preferred, any appropriate method may be used, such as mixing the various components, provided the cores are dispersed in the resultant matrix and do not form agglomerates.

The tool components of the invention can be used in a wide range of applications, but find particular application in drill bit applications, typically in roller cone bits and drag bits.

The composite material manufactured in accordance with the invention has several advantages over the prior art materials described earlier. The carbide cores are bonded to the PCD matrix via a strong mechanical keying arrangement, thus overcoming the problem of weak bonding of individual diamond particles to a cobalt matrix. Due to the properties mismatch between carbide and PCD, the carbide cores will be in a state of tension, while the PCD matrix will be in compression. The compressive stresses experienced by the PCD continuous matrix will increase the strength of the resulting material, compared to the strength of conventional PCD material. The carbide cores will be in tension. Cracks propagating through this material will be attracted to these regions. If the carbide grade used is a particularly tough one, then the propagation of this crack through this phase will be energetically costly. Thus crack propagation within the resulting material will be more difficult than it would be within conventional PCD. By using granules to create the composite carbide - PCD material, the danger of generating agglomerates of the carbide phase, or very large continuous fillets of PCD, are largely avoided. This allows for a tighter control of the defect size in such materials, thus ensuring more reproducible properties for them.

The combination of these advantages provides a material with good toughness, strength and wear resistance. These critical properties can be tailored to lie between those of substrate and PCD. By varying the grade of

- 12 -

PCD as well as the volume fraction of this component in the composite cermet, both the wear resistance, as well as the elastic properties and the thermal expansion coefficient of the resulting composite can be varied to suit the purpose of a particular drill insert design.

Where the composite material of the invention is used as an interlayer, it allows for the production of tool components with much thicker PCD layers, due to the reduced stresses at the interface between the PCD layer and the intermediate layer as compared to the much higher stresses at the interface between the PCD layer and the substrate of a conventional tool component.

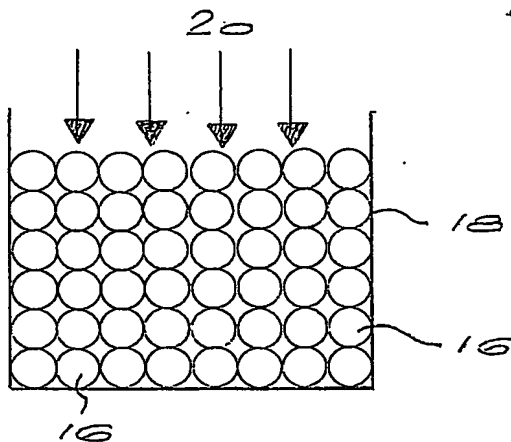
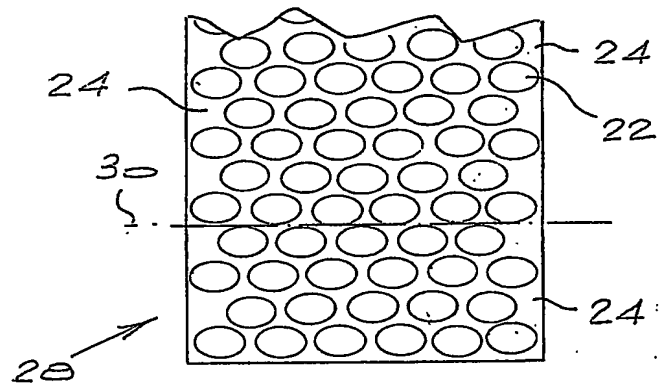
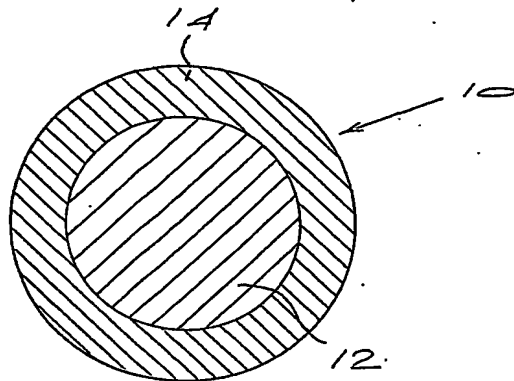
DATED THIS 29th DAY OF OCTOBER 2002



SPOOR & FISHER

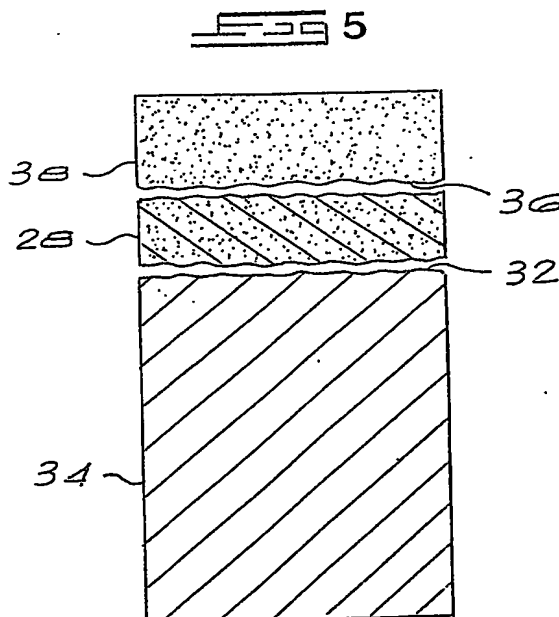
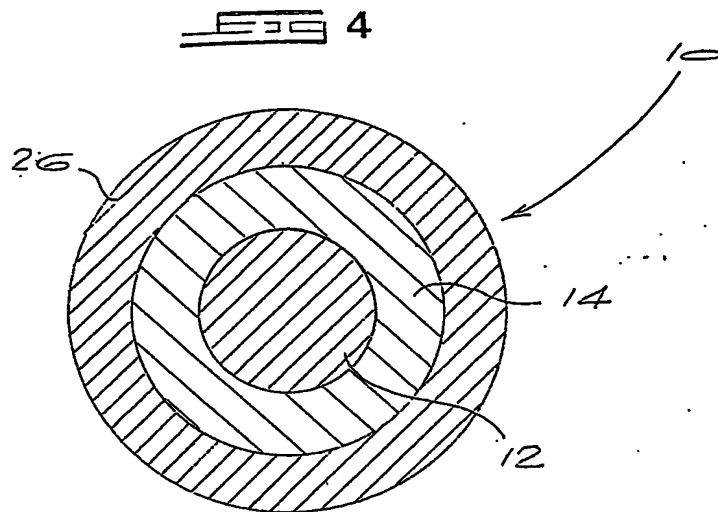
APPLICANT'S PATENT ATTORNEYS

2002/831




SPOOR & FISHER
Applicant's Patent Attorneys

2002/8731



[Signature]
SPOOR & FISHER
Applicant's Patent Attorneys

SPOOR & FISHER

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
LATE LODGING OF DOCUMENTS
(Section 30(6) - Regulation 38)

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REPUBLIC OF SOUTH AFRICA
REVENUE

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REPUBLIC OF SOUTH AFRICA

OFFICIAL APPLICATION NO.

LODGING DATE

21 01 2002/8731

22 29 OCTOBER 2002

PA133718/P

FULL NAME(S) OF APPLICANT(S)

71 ELEMENT SIX (PROPRIETARY) LIMITED

ADDRESS FOR SERVICE

74 SPOOR & FISHER, SANDTON

The following documents not accompanying the application as lodged or required by the Registrar are hereby submitted:

- ☐ 1. A single copy of a provisional specification of pages.
- ☐ 2. Drawings of
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☒ 5. Assignment of invention.
- ☐ 6. Certified priority document.
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Dated: 2-Dec-2002

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PATENT ATTORNEYS FOR THE APPLICANTS

ACKNOWLEDGEMENT OF LATE LODGING OF DOCUMENTS

REGISTRAR OF PATENTS

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| 2002-12-03 |
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